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Pressure-induced property improvement of magnesium diboride wire

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Abstract. The 4.2 K non-barrier transport J_c values of the powder-in-tube (PIT) *in-situ* MgB_2 wires have been enhanced by cold high pressure densification (CHPD). With respect to the control wire, the 1.5 GPa pressure induced the J_c improvement of the wire at 4.2 K and 10 T by 25 %. The J_c enhancement induced by the CHPD may result from two aspects. First, the CHPD resulted in the reduction of the transverse MgB_2 core area. Second, the grain connectivity of the PIT *in-situ* MgB_2 wire was improved by the CHPD, which is reflected by the reduced porosity.

1. Introduction

Compacted-powder pellets and the cores of powder-in-tube (PIT) wires are inherently porous. Thus the packing densities of sintered *ex-situ* processed wires, based geometrically on the packing density of a uniform-sized starting powder fill, would be around 65% (although powders of mixed sizes will fill space more efficiently). The expected density of *in-situ*-processed material is complicated by shrinkage associated with the $Mg+2B \rightarrow MgB_2$ reaction. As a result it can be shown that a mixture of equi-sized Mg and B particles will after reaction lead to an MgB_2 density of 49%, although the use of coarse Mg particles could raise the final density to 62% [1]. Porosity in the starting powder fill (and staring porosity plus reaction in the case of *in-situ* material) impedes current flow and prevents the J_c of the bulk or wire from attaining its intrinsic intragranular value. Current transport between grains may be reduced by the presence of insulating partial coatings [2]. These effects can combine to yield very low connectivities [3], for example 20~40% [1] which would offer the possibility of transport J_c increases by factors of 5~2.5. The problems of porosity and intergranular coatings call for separate solutions. Many researchers have addressed the former and, as reviewed below, have attempted to squeeze out porosity by applying high pressures to bulk and wire samples both during the reaction heat treatment as well as prior to it.

Shields *et al.* [4] reported that hot isostatic pressing (HIP) induced the magnetic critical current densities as high as 1.3×10^6 A/cm^2 at 0 T and 9.3×10^5 A/cm^2 at 1 T of the MgB_2 bulks at 100 MPa and 950 ºC, 20 K. Serquis *et al.* [5] presented that the J_c values of HIP – processed PIT MgB_2 wires reached 10^6 A/cm^2 at 5 K and 0 T and the order of 10^4 A/cm^2 at 1.5 T and 26.5 K. Susner *et al*. [6] reported that a MgB_2 monofilamentary strand hot-pressed by 458 MPa achieved the J_c value of 1.81×10^5 A/cm^2 at 4.2 K and 5 T, with respect to 0.86×10^5 A/cm^2 of the unpressed strand.

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Cold high pressure densification (CHPD) has been considered as a method for improving the grain connectivity of the PIT *in-situ* MgB_2 wire as evidenced by the enhancements of relative mass density, electrical resistivity and transport J_c [7, 8]. Flükiger and Hossain [7] indicated that the CHPD induced the J_c improvement by 53 % at 4.2 K and 10 T for a binary Fe/MgB_2 round wire. Flükiger *et al.* [9] also presented that the J_c value of monofilamentary *in situ* MgB_2 wire doped with $C_4H_6O_5$, heat-treated with 4.0 h/600 °C, and cold-densified by the pressure of 1.5 GPa reached about 4.3×10^4 A/cm^2 at 4.2 K and 10 T, with respect to 2.2×10^4 A/cm^2 of the control wire. In this paper, the influence of the CHPD on the current-carrying capacity of the 2 % - C doped PIT *in-situ* MgB_2 wires was investigated. The MgB_2 wire heat-treated by 1.0 h/675 °C and cold-densified by the pressure of 1.5 GPa was successfully prepared with 4.2 K non-barrier transport J_c value of 3.6×10^4 A/cm^2 at 10 T and the order of 10^5 A/cm^2 at 7 T.

2. Experimental

2.1 Sample Preparation

Several coils of 2 % carbon-doped unreacted PIT *in-situ* MgB_2 wires were manufactured and provided by the Hyper Tech Research, Inc. The outer sheath and chemical barrier of the MgB_2 wire are copper and niobium, respectively. The outer diameter of the MgB_2 wire is 0.834 mm; the fill factor of MgB_2 core is 25.7 % and the ratio of Mg:B is 1:2.

The wires to be pressed were held between the jaws of a six-inch Kurt vice. The jaws were faced with blocks of heat treated M2 steel and plates of tungsten carbide as shown in Figure 1. Held between this assembly was the wire to be pressed and upper and lower blades of tool steel 0.75 mm thick. Also located between the jaws was a 10 ton hydraulic short-body ram. Vertical pressure was applied to the tool-steel blades, and hence the subject wire, by a 20-ton hydraulic ram. Vertical pressures of 1.0, 1.5, and 2.0 GPa were applied to the wires which at the same time were exposed to 2.0 GPa horizontal pressure, Table 1.

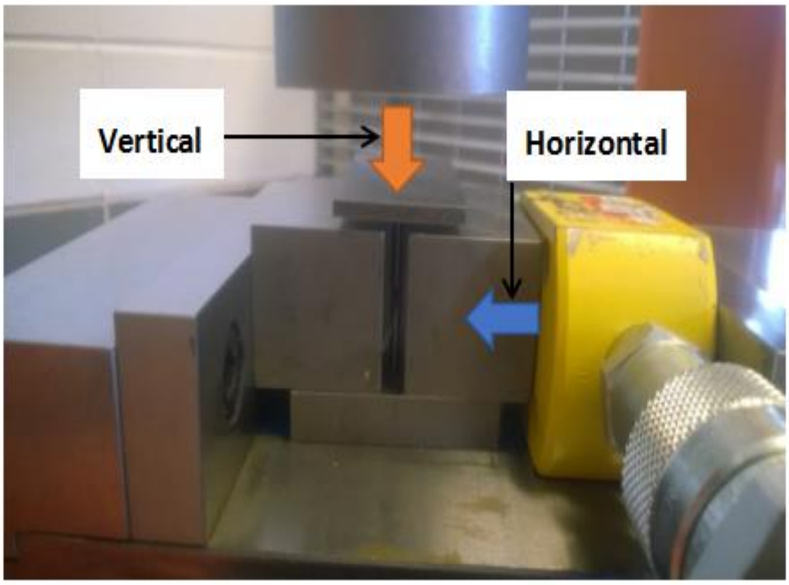

Figure 1. The assembly used for applying pressures on the MgB_2 wires. The directions of vertical pressure and horizontal pressure were defined.

IOP Conf. Series: Materials Science and Engineering **279** (2017) 012024 doi:10.1088/1757-899X/279/1/012024

During vertical pressing there is a tendency for the wires to expand horizontally and to extrude past the blades as shown in Figure 2.

A fixed 2.0 GPa horizontal pressure continually applied prevented this from happening and enabled clean rectangular cross sections to be maintained, Figure 3.

All the wires were encapsulated in quartz tubes under 200 torr Ar and heat treated for 1h/675^{o}C.

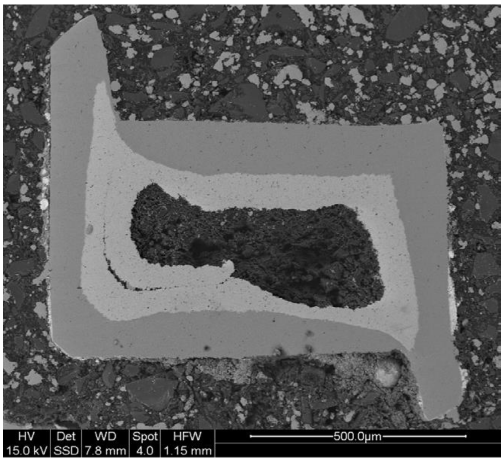

Figure 2. The extrusion of the wire vertically cold-pressed by 2.0 GPa.

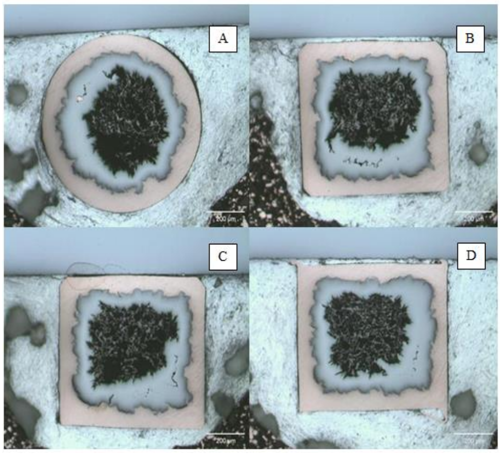

Figure 3. Transverse cross sectional areas of the MgB_2 wires (A) W00 (B) W10 (C) W15 (D) W20.

Table 1: Pressure schedule for MgB_2 wire

Sample No.	Vertical Pressure (GPa)	Horizontal Pressure (GPa)
W00	0.0	0.0
W10	1.0	2.0
W15	1.5	2.0
W20	2.0	2.0

2.2 Measurement

The transport critical current densities of the wires were measured as a function of applied magnetic fields up to 12 T at 4.2 K in a pool of boiling liquid helium by the four-probe technique. The wires were 5 cm long, the gauge length between voltage taps was 5 mm, and the voltage criterion was 1.0 μV/cm.

3. Result and Discussion

3.1. Transport Result

Figure 4 shows the 4.2 K non-barrier transport critical current densities of the cold-pressed and control wires as a function of applied magnetic field up to 12 T. All cold-densified wires have J_c values above 3.0×10^4 A/cm^2 at 10 T. Compared with control wire, the 4.2 K non-barrier transport J_c values of W10

(1.0 GPa cold-pressed wire) and W15 (1.5 GPa cold-pressed wire) have been enhanced at all applied magnetic fields; for example, the 4.2 K, 10 T non-barrier transport J_c values of the W10 and W15 are 15 % and 25 % higher than that of W00. The best J_c values were obtained by the wire W15 at all applied magnetic fields and are found to be 3.6×10^4 A/cm^2 at 10 T and 1.1×10^5 A/cm^2 at 7 T.

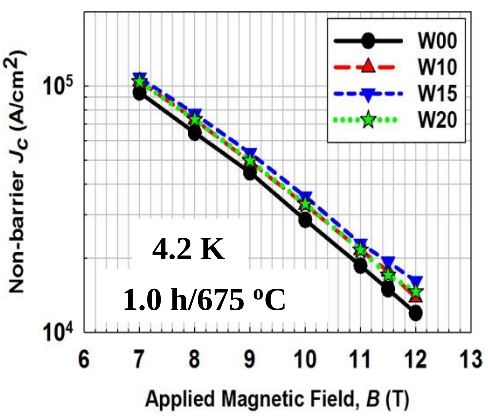

Figure 4. Non-barrier transport critical current density J_c of the MgB_2 wires as a function of applied magnetic field at 4.2 K.

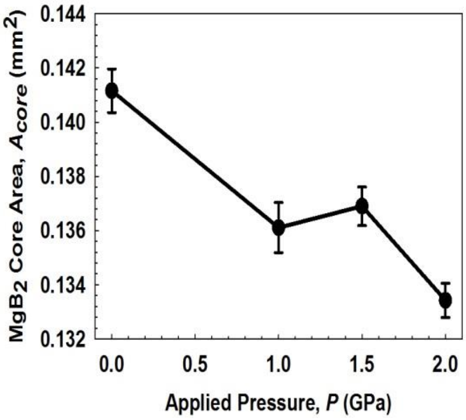

Figure 5. Average transverse MgB_2 core area of the PIT *in-situ* MgB_2 wire with error bars versus applied pressure.

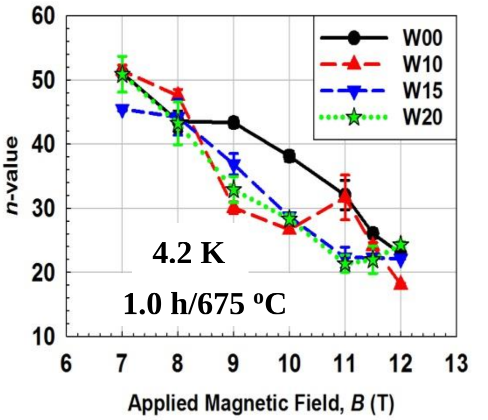

Figure 6. Average *n*-values of the MgB_2 wires with error bars as a function of applied magnetic field at 4.2 K.

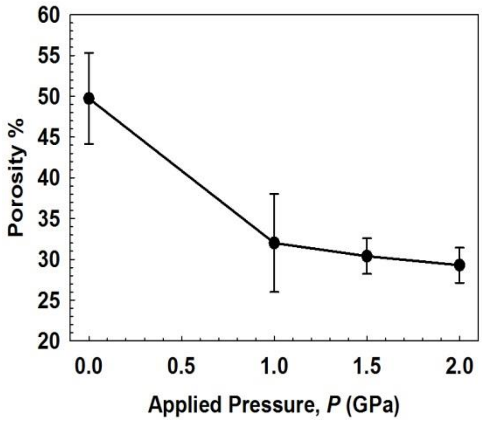

Figure 7. Average porosity of the PIT *in-situ* MgB_2 wire with error bar versus applied pressure.

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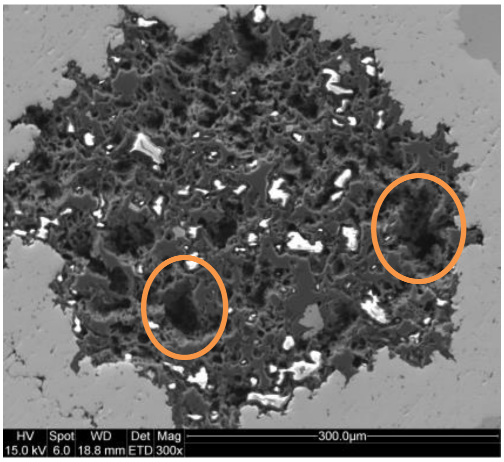

Figure 8. The SEM image in SE mode of the transverse MgB_2 core area of the W00. Two large voids were marked in this figure.

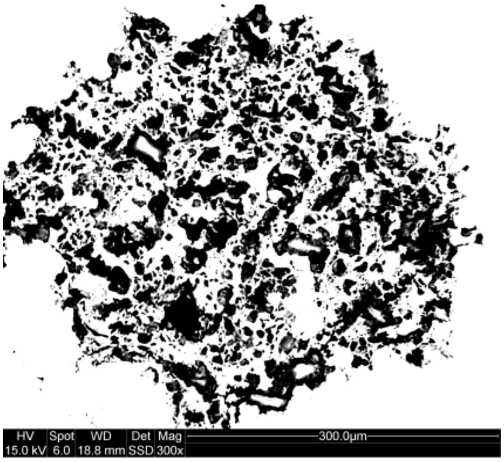

Figure 9. The SEM image in BSE mode of the transverse MgB_2 core area of the W00.

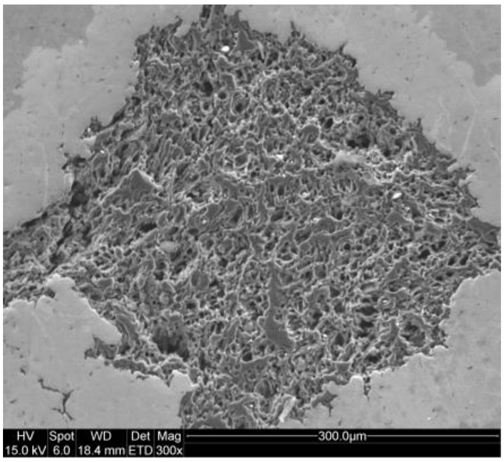

Figure 10. The SEM image in SE mode of the transverse MgB_2 core area of the W15.

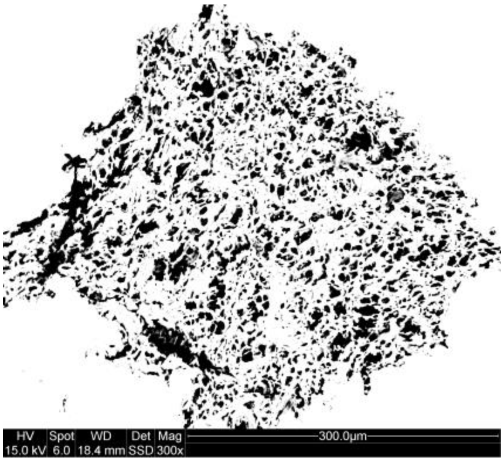

Figure 11. The SEM image in BSE mode of the transverse MgB_2 core area of the W15.

The J_c enhancements of the cold-pressed MgB_2 wires are partially due to the reduction of the transverse MgB_2 core area induced by the CHPD as shown in Figure 5. The pressure of 2.0 GPa led to the largest reduction of the transverse MgB_2 and was found to be 5.5 %. If the reduced transverse MgB_2 core area is the unique factor to inducing the J_c enhancements of the MgB_2 wires, the J_c of W20 will be improved by a factor no higher than 5.8 %. However, the J_c enhancement of W20 was at least 10.4 % at the range of 7 to 12 T indicating that the CHPD has the potential of improving other properties of the PIT *in-situ* MgB_2 wire as well as reducing transverse MgB_2 core area leading to the enhancements of its J_c values. It can be seen that the 4.2 K non-barrier transport J_c values of W20 were lower than those of the W15 at all applied magnetic fields suggesting the too large pressure might introduce defects in the MgB_2 wire and therefore induced the J_c degradation of the MgB_2 wire. W20 had the 4.2 K non-barrier transport J_c values of 3.4×10^4 A/cm^2 and 1.1×10^5 A/cm^2 at 10 T and 7 T, respectively, which were still higher than those of W00. Figure 6 shows the 4.2 K *n*-values of the cold-densified and control wires as a function of applied magnetic field up to 12 T. The largest error in the measurement of *n*-value is 11%. At 10 T the cold-densified wire has a 34% decrease in *n*-value, but the *n*-value of W00 is close to those of the cold-densified wires at 8 T. The field dependence of *n*-value suggests that the CHPD might not induce higher level of extrinsic defects in PIT *in-situ* MgB_2 wire.

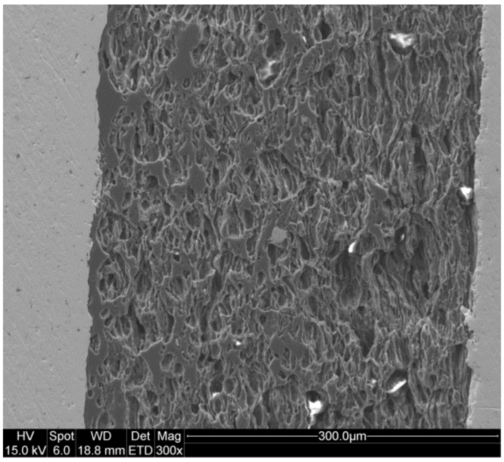

Figure 12. The longitudinal cross section area of W00.

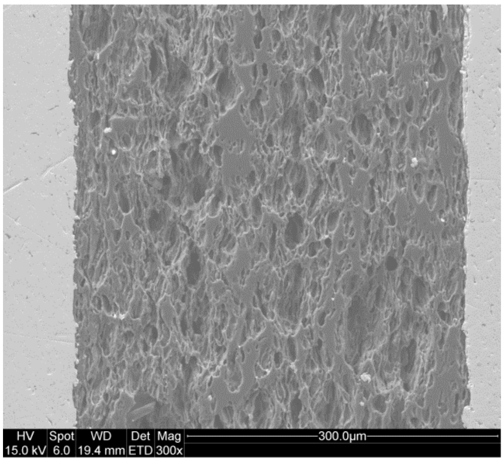

Figure 13. The longitudinal cross section area of W15.

3.2. Microstructures
Figure 7 shows the average porosity of the wire versus the applied pressure. It can be seen that the wire processed by the CHPD have lower porosity than the control wire. Figure 8 - Figure 11 demonstrate the SEM images with secondary - electrons (SE) and back – scattered electrons (BSE) modes of the transverse MgB_2 core area of W00 and W15. In the BSE SEM image as in Figure 9 and Figure 11, the dark parts represent the voids existing in the wires and light parts represent the MgB_2 stringers in the wires. The SEM images in BSE mode were taken under same brightness and contrast. Consequently, the porosity of the wire can be obtained by calculating the area fraction of the dark parts in the BSE mode. The SEM images in SE mode as shown in Figure 8 and Figure 10, there exists some large voids in W00, but no large void was observed in W15. Based on these results, it can be concluded the CHPD has the ability to squeeze out the porosity of the PIT *in-situ* MgB_2 wire suggesting the enhanced grain connectivity of the MgB_2 wire. On the other hand, the CHPD has the limited ability to further reduce the porosity of the wire when the applied pressure was larger than 1.0 GPa. For example, the reduction of the average porosity of the MgB_2 wire was only 8.5 % when the applied pressure was increased from 1.0 GPa to 2.0 GPa; however, it was reduced by 35.6 % when the applied pressure was increased from 0.0 GPa to 1.0 GPa. The longitudinal cross section areas of W00 and W15 are shown in Figure 12 and Figure 13. Susner *et al.* [10] described that continuous MgB_2 stringers in PIT *in-situ* wire were aligned along drawing direction and elongated voids which were left

behind by melted Mg powder. It can be seen that the CHPD does not alter the morphology of the continuous MgB_2 stringers in the PIT *in-situ* MgB_2 wire.

4. Conclusions

Cold high pressure densification can improve the 4.2 K non-barrier transport J_c of the PIT *in-situ* MgB_2 wire in present paper. The best 4.2 K non-barrier transport J_c performance was obtained by W15 at all applied magnetic fields. The *n*-values of all cold-densified wires are above 30 when the applied magnetic field is below 9 T. The pressure dependence of the porosity of the PIT *in-situ* MgB_2 wire was discussed. The reduced porosity of the MgB_2 wire processed by the CHPD reflects that the J_c enhancement induced by the CHPD might result from the improved grain connectivity of the PIT *in-situ* MgB_2 wires.

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